



Distribution of residual strain around nanoindentations in silicon



X. Li ^{a,b}, Z. Li ^a, X.F. Tao ^{b,*}, L.L. Ren ^b, S.T. Gao ^b, G.F. Xu ^a

^a School of Materials Science and Engineering, Central South University, Changsha 410083, China

^b Division of Nano Metrology and Materials Measurement, National Institute of Metrology, Beijing 100013, China

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ABSTRACT

Residual strain around pyramidal nanoindentations on single-crystal silicon is mapped by electronic backscatter diffraction system and CrossCourt software. Both of the (001) and (111) planes present anisotropic strain features adjacent to indentations in specific crystallographic orientations with strain resolutions of 2.2×10^{-4} and 2.5×10^{-4} for Si (001) and (111) surfaces, respectively. The anisotropic distribution of strain is due to the occurrence of dislocation slip in the silicon microstructure, which is determined by the number and type of slip systems and corresponding Schmid factor. The nanoindentation plastic deformation of single-crystal silicon is a process accompanied by dislocation slip on specific planes in specific directions.

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1. Introduction

The need to know local residual strain (ε) states in materials is of the utmost importance in determining the performance and productivity of some silicon (Si)-based devices and structural components [1]. Strain dramatically increases the mobility of carriers and leads to significantly enhanced performances in field effect transistors [2]. In optical excitation, the presence of residual strain in Si substrate changes the structure of photonic quantum-well and thus influences the output color and lifetime of visible light-emitting and laser diodes [3]. However, in various isolation structures, small strain is propitious to prevent the generation of dislocations which can destructively degrade the device response [4]. Different manufacturing techniques and service conditions will generate various residual strain states. Hence, the desire to measure and map the distribution of residual strain is pervasive in a number of technology areas [5].

Instrumented indentation has been extensively used in measuring mechanical properties of Si-based devices and to evaluate the reliability of these devices as a model of contact flaws [6]. The indentation deformation of Si materials is accomplished by dislocation movement and phase transformation [7]. Residual stress states around these deformation zones were studied by X-ray diffraction and Raman spectroscopy in the scale of micron meters [8,9]. Electron backscattered diffraction (EBSD) is a versatile technique that has shown great promise for measuring residual strain or stress on the surface of crystalline materials with high

resolution [10]. Based on the application of Raman microscopy and EBSD technique in measuring the lattice distortion of single-crystal Si, the stress distribution at sub-micron scale around a 350 mN wedge indentation ($30 \mu\text{m} \times 350 \mu\text{m}$) was revealed [11]. On different Si crystallographic planes, the residual stress states of a smaller $12.5 \mu\text{m} \times 12.5 \mu\text{m}$ field with 80 mN central indentations were mapped by confocal Raman, which obviously revealed anisotropic stress patterns with an orientation specific symmetry [12]. However, the development of advanced micro-nano Si-based devices indicates that residual strain/stress distribution in Si materials needs to be studied at a smaller field and, where higher spatial and strain resolutions are required. In this work, residual strain of diamond cubic Si induced by 10 mN pyramidal nanoindentations was two-dimensionally mapped by using high resolution EBSD detector and CrossCourt software, where all strain components were displayed. We dedicated to mapping strains at smaller peak load and scanning step, which leads to higher spatial and strain resolutions. Furthermore, the anisotropic characteristics of residual strain were explained with dislocation mechanisms.

2. Materials and method

The test samples in this work were $200 \mu\text{m}$ thick \times 3 mm diameter, polished Si(001) and Si(111) crystallographic planes using ion milling. The samples were indented using a Berkovich indenter with a tip curvature radius of 100 nm approximately in a clean room environment (class 10^3). The pyramidal indenter was put perpendicular to the Si(001) and Si(111). During indentation experiments, both of the loading and unloading rates were 2 mN s^{-1} . The load was linearly increased with time to the

* Corresponding author.

E-mail address: taoxf@nim.ac.cn (X.F. Tao).

maximum value of 10 mN, kept for 2 s, and linearly decreased. A Nordlys Nano EBSD detector (1344×1024 pixels) assembled in a Zeiss Ultra 55 field-emission scanning electron microscope (FE-SEM) was used to acquire the EBSD patterns of regions adjacent to indentations. The microscope worked at an acceleration voltage of 20 kV, a scanning step of 50 nm and a stage drift less than 12.5 nm/min. The pattern acquisition time of a $2.8 \mu\text{m} \times 2.8 \mu\text{m}$ area containing central indentation was 10 min and thus the total

image drift is about 125 nm. Considering the scan size and step size, the drift is acceptable and insignificant for the final strain mapping. The obtained EBSD patterns were analyzed using CrossCourt 3 software which has a strain sensitivity of $\pm 10^{-4}$ and a misorientation sensitivity of $\pm 0.006^\circ$ for calculating the residual strain [13]. The key of CrossCourt is the use of cross-correlation analysis to determine pattern shifts, i.e., relative positions of common diffraction contrast features in the test EBSD pattern

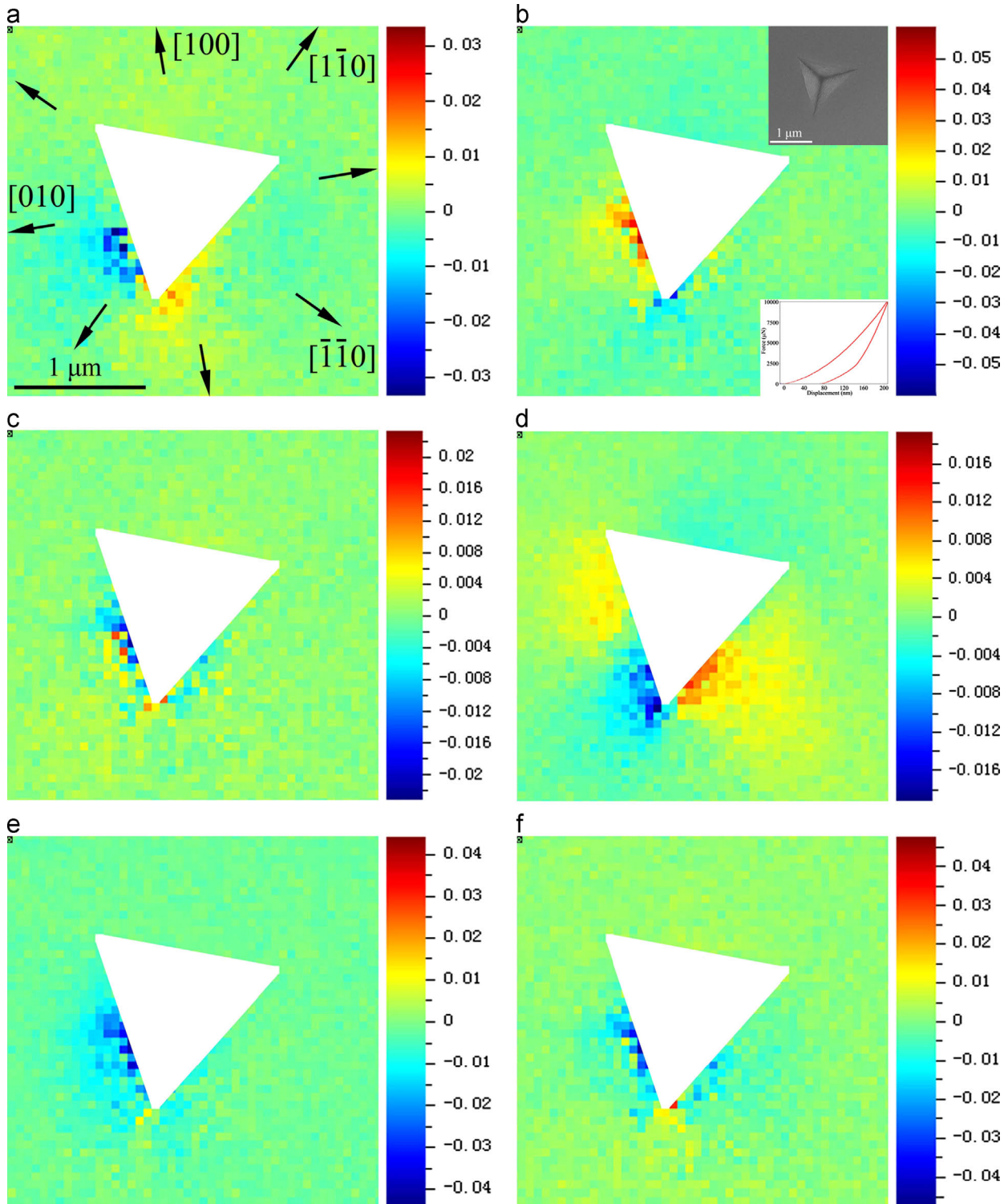


Fig. 1. Residual strain mapped by EBSD of Si sample indented perpendicular to Si(001): (a) ϵ_{11} ; (b) ϵ_{22} , the insets are corresponding load–displacement curve and SEM image; (c) ϵ_{33} ; (d) ϵ_{12} ; (e) ϵ_{13} ; (f) ϵ_{23} . Scan sizes: $2.8 \mu\text{m} \times 2.8 \mu\text{m}$. The EBSD pattern of black spot is the reference.

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